



Effect of Transmission Electron Microscopy Sample Preparation Methods on Nano Structure and Properties of Metallic Glasses

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Background Information

- Transmission Electron Microscope (TEM) examines the atomic structure of materials by transmitting an electron beam through a sample.
- Diffraction pattern is created with the interaction of an electron beam with the crystal plane. The planes of a crystal would diffract electrons if these planes were lying approximately parallel to the electron beam. [1]
- MGs are metallic materials with disordered atomic structure, and they show many important properties that are not observed in crystalline metals and glasses. [2] For instance, because MGs relatively lack crystalline phases compared to metallic materials, they also lack common structural defects, such as dislocations and grain boundaries, which decrease the theoretical yield strength of crystalline materials. In this research, $\text{Ti}_{48}\text{Zr}_{20}\text{Be}_{15}\text{V}_{12}\text{Cu}_5$ was used as the metallic glass sample in this research.

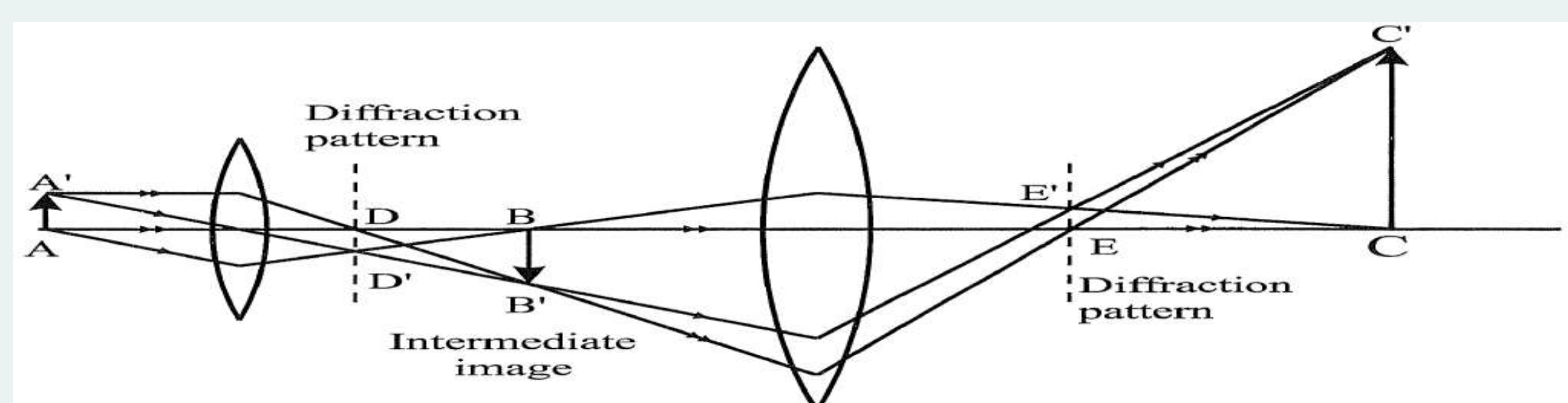


Figure 1: The ray diagram of a two stage projection microscope showing the position of the diffraction pattern and image.[1]

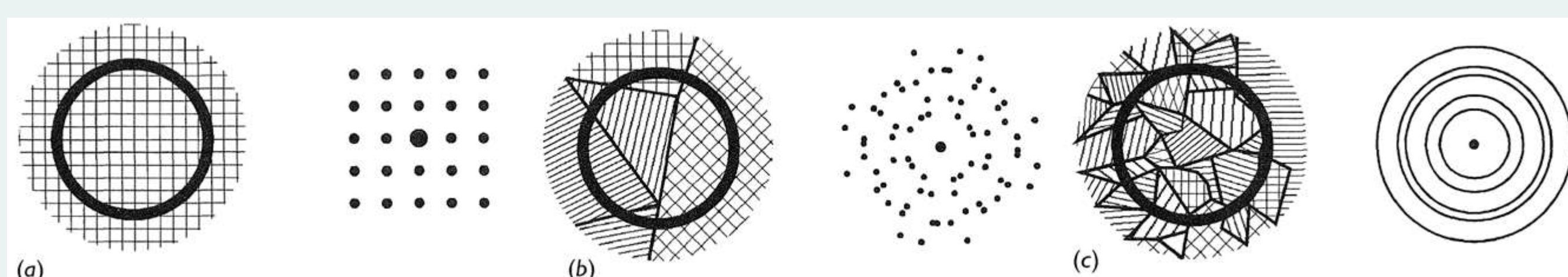


Figure 2: Types of diffraction pattern which arise from different specimen microstructures. (a) A single perfect crystal. (b) small number of grains (c) a large number of grains. [1]

Conclusion

- (1) The metallic glass samples prepared by FIB showed no apparent damage to the sample.
- (2) Mechanically polished sample showed the signs of plastic deformation during the polishing process. Thus while this method is not suitable for studying the structure, but it may be useful for observing the mechanical deformation behavior of metallic glasses and glass composites.
- (3) Electropolished samples were too thick to generate any diffraction signals. When compared to other two methods, it was much more difficult to control the electropolishing conditions and the sample quality.

Based on these results, we can conclude that FIB may be the best method for observing the structure of MG. FIB ensures fast and reliable sample preparation process, and yields fairly good quality samples.

Future Work

1. Creating MG sample which is thin enough by electro polishing in order to compare the quality of the sample to others samples prepared by two other methods.
2. FIB may induce small surface damage, but the extent of damage is difficult to quantify in amorphous materials. We will use FEM to check if there is any measurable change in the structure due to FIB damage.
3. Using the new sample preparation methods that we develop here, the structure at the interface between the glass and the crystal particles will be examined. This will provide useful insights on the superb mechanical properties of metallic glass composites.

Sample Preparation Methods

1. Mechanical polishing

This method is based on surface polishing of materials using diamond embedded lapping films on a disk polishing machine. By polishing both sides of the sample until they become atomically smooth with a small wedge angle (typically 2-4°), the tip of the sample wedge can become as thin as ~10 nm.

2. Electropolishing

This method is based on acid etching of the sample in an electrically biased environment. When the sample gets etched, the sample gets gradually thinner until a hole is formed, with thin sample areas around it.

3. Focused Ion Beam (FIB)

FIB uses a Ga ion beam, which can be focused on a nanoscale area, and therefore any nanoscale region of the material can be selected and prepared for TEM observation.



Figure 3: (a) Allied High Tech mechanical polisher, (b) Fischione Twin-jet electropolisher, and (c) FEI Helios FIB that will be used in this project

Result

1. MG sample prepared by FIB

The STEM image shows two distinctive regions with clear difference in their contrast. Nanodiffraction analysis showed that the brighter regions have glass structure, while the darker regions have crystal structure. The glass structure appears to be quite homogeneous, as indicated by the pattern in (b) which shows no apparent azimuthal variation in intensity.

2. MG sample prepared by mechanical polishing

The regions without any contrast, the area inside the yellow box, are amorphous. Also, the regions with brighter stripes that red arrow indicates are crystals. The crystalline phase had bright lines that are not present in the FIB sample. The structure of the glass phase also shows more inhomogeneous structure, as indicated by larger speckles in the pattern. (white circles in Figure 4 (b)).

3. MG sample prepared by electropolishing

Failed to create a thin MG sample by electropolishing.

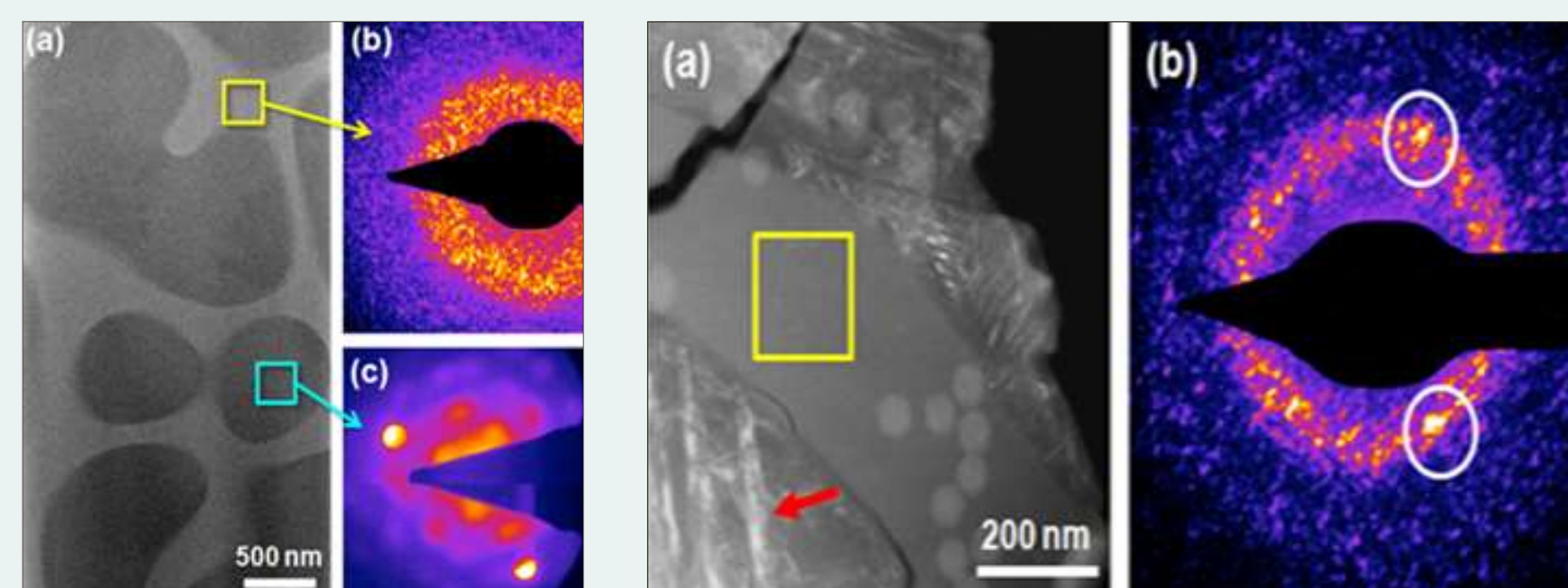


Figure 4: STEM image and nanodiffraction pattern of metallic glass sample prepared by FIB (left) and mechanical polishing (right).

References

- [1] Humphreys, John, and Richard Beanland. *Electron Microscopy And Analysis*. By Goodhw J. Peter. 3rd ed. N.p.: n.p., 2001. 40-65. Print.
- [2] A.L. Greer, Metallic glasses...on the threshold. *Materials Today*, **12**(1-2), 14-22 (2009).

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